भारतीय मानक
Indian Standard

IS 16713 : 2018

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Reclaimed Isobutene-Isoprene (IIR) Rubber — Evaluation Procedure

ICS 83.040.10

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July 2017 **Price Group 2**

Method of Test for Rubber and Rubber Products Sectional Committee, PCD 29

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Method of Test for Rubber and Rubber Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard is based on ISO/TS 16096 : 2014 'Reclaimed rubber derived from products containing mainly natural rubber — Evaluation procedure'.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*).' The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

RECLAIMED ISOBUTENE-ISOPRENE (IIR) RUBBER — EVALUATION PROCEDURE

1 SCOPE

This standard specifies:

- the physical and chemical tests on raw reclaimedisobutene-isoprene (IIR) rubber; and
- standard materials, standard test formulations, equipment, and processing methods forevaluating the vulcanization characteristics, and the mechanical properties of reclaimed isobutene-isoprene (IIR) rubber.

2 REFERENCES

The following standards contain provisions which,through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below.

IS/ISO No.	*Title*
3400 (Part 1) : 2012/ ISO 37 : 2011	Methods of test for vulcanized rubbers: Part 1 Tensile stress-strain properties (*third revision*)
3400 (Part 23/ Sec 1)/ISO 7619-1: 2010	Methods of test for vulcanized rubbers : Part 23 Rubber — Determination of indentation hardness by means of pocket hardness meters, Section 1 Durometer method (Shore hardness) (*first revision*)
3660 (Part 7) : 2013/ ISO 289-1:2005	Methods of test for natural rubber: Part 7 Determination of Mooney viscosity (NR:8) (*third revision*)
3708 (Part 9) : 2005/ ISO 247 : 1990	Methods of test for natural rubber latex: Part 9 Determination of total ash (NR 16) (*second revision*)
11720 (Part 2) : 2013/ ISO 3417 :2008	Methods of test for synthetic rubber : Part 2 Measurement of vulcanization characteristics with the oscillating disc curemeter
ISO 1407 : 2011	Rubber — Determination of solvent extract
ISO 1408 : 1995	Rubber — Determination of carbon black content — Pyrolytic and chemical degradation methods
ISO 1795 : 2017	Rubber, raw natural and raw synthetic — Sampling and further preparative procedures
ISO 2393 : 2014	Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures
ISO 6502	Rubber — Guide to the use of curemeters (*under formulation*)
ISO 23529 : 2016	Rubber — General procedures for preparing and conditioning test pieces for physical test methods
ASTM D297-13	Standard test methods for rubber products — Chemical analysis

3 SAMPLING AND SAMPLE PREPARATION

3.1 Take a laboratory sample of approximately 1.5 kg by the method described in ISO 1795.

3.2 Prepare the test sample in accordance with ISO 1795.

4 PHYSICAL AND CHEMICAL TESTS ON RAW RUBBER

4.1 Mooney Viscosity

Determine the Mooney viscosity in accordance with IS 3660 (Part 7) on a test piece prepared as indicated in **3.2**.

Record the result as ML (1 + 4) at 100 °C.

4.2 Acetone Extract

Determine the acetone extract in accordance with Method A or Method B of ISO 1407.

4.3 Ash

Determine the ash content in accordance with Method A of IS 3708 (Part 9).

4.4 Carbon Black

Determine the carbon black content in accordance with ISO 1408, Method A.

4.5 Rubber Content

Determine the rubber content, R (in %), in accordance with ASTM D297-13, Part A, Clauses 11, 12, and 13.

$$R = 100 - (a + b + c)$$

where

a = the carbon black content, in percent;
b = the ash content, in percent; and
c = the acetone extract, in percent.

5 PREPARATION OF TEST MIX FOR EVALUATION

5.1 Standard Test Formulation

The standard test formulation for evaluation of reclaimed isobutene-isoprene (IIR) rubber is given in Table 1.

The materials used shall be national or International Standard reference materials. If no standard reference material is available, the materials to be used shall be agreed between the interested parties.

Table 1 Standard Test Formulation
(*Clause* 5.1)

Material (1)	Parts by Mass (2)
Reclaimed isobutene-isoprene (IIR) rubber	$100.00 + x + y + z$
Zinc oxide[a]	5.00
Sulfur[a]	2.00
ZBEC (zinc dibenzyldithiocarbamate)[a]	1.50
Mercaptobenzothiazole (MBT)	1.00

NOTES
1 x is the number of parts of carbon black to 100 parts of rubber in reclaimed isobutene-isoprene rubber.
2 y is the number of parts of acetone extract to 100 parts of rubber in reclaimed isobutene-isoprene rubber.
3 z is the number of parts of ash to 100 parts of rubber in reclaimed isobutene-isoprene rubber.
4 a is powder materials used (standard curing ingredients used in the industry).

5.2 Mixing Procedure — Mixing with a Laboratory Mill

The standard laboratory mill-batch mass, in g, shall be enough to form proper rolling band. The surfacetemperature of the rolls shall be maintained at 50 °C ± 5 °C throughout the mixing.

A good rolling bank at the nip of the rolls shall be maintained during mixing. If this is notobtained withthe nip settings specified hereunder, small adjustments to the mill openings can be necessary.

A mill batch mass based on two times the formulation mass can also be used. However in this case, moreadjustment to the mill openings will be necessary.

		Duration min	*Cumulative Time* min
a)	Band the reclaimed rubber with the mill opening set at 1.2 mm.	1.0	1.0
b)	Add the zinc oxide across the mill rolls at a uniform rate. When all the zinc oxide has been incorporated, make a 3/4 cut from each side. Do not cut the batch while powder is evident in the bank or on the milling surface. Be certain to return to the batch any materials that drop through the mill	1.0	2.0
c)	Add the MBT across the mill rolls at a uniform rate. When all the powder has been incorporated, make a 3/4 cut from each side.	1.0	3.0
d)	Add the ZBEC across the mill rolls at a uniform rate. When all the powder has been incorporated, make a 3/4 cut from each side.	1.0	4.0
e)	Add the sulfur across the mill rolls at a uniform rate. When all the sulfur has been incorporated, make a 3/4 cut from each side.	1.0	5.0
f)	Cut the batch from the mill and take off the mill rolls.	2.0	7.0
g)	Set the mill opening to 1.2 mm and pass the rolled batch end wise through the mill 12 times.	3.0	10.0
	Total time	**10.0**	

h) Sheet the batch in grain direction to an approximate thickness of 6 mm and check-weigh thebatch (*see* ISO 2393). If the mass of the batch differs from the theoretical value by more than + 0.5 percent or-1.5 percent, discard the batch and re-mix.

j) Remove sufficient material for curemeter testing.

k) Sheet the batch to approximately 2.2 mm for preparing test slabs or to theappropriate thickness for preparing ring or dumbbell test pieces in accordance with IS 3400 (Part 1).

m) After mixing, condition the batch for at least 2 h, but not more than 24 h, if possible at standard laboratory temperature and humidity as defined in ISO 23529.

NOTE — For very low Mooney viscosity reclaim rubber, tendency of reclaim sticking to the roll can be observed. Hence, cutting as mentioned in the procedure can not be possible. In such case, scraper blade as shown in Fig. 1 can be used. After scraping the material each time, it should be folded two times and passed through mill again to ensure proper blending of chemical.

6 EVALUATION OF VULCANIZATION CHARACTERISTICS BY A CUREMETER TEST

6.1 Using an Oscillating Disc Curemeter

Measure the following standard test parameters:

M_L, M_H at defined time,t_{s1}, $t'_c(50)$, $t'_c(90)$, and $t'_c(95)$

in accordance with IS11720 (Part 2), using the following test conditions:

- oscillation frequency: 1.7 Hz (100 cycles per minute)
- amplitude of oscillation: 1° of arc (An amplitude of oscillation of 3° of arc is permitted as an alternative. If such an amplitude is chosen, measure t_{s2} instead of t_{s1})
- torque range: to be chosen to give at least 75 percent of full-scale deflection at M_H
- die temperature: 160 °C ± 0.3 °C
- pre-heat time: None.

6.2 Using a Rotorless Curemeter

Measure the following standard test parameters:

F_L, F_{max} at defined time,t_{s1}, $t'_c(50)$, $t'_c(90)$, $t'_c(95)$ and t'_c (99)

in accordance with ISO 6502, using the following test conditions:

- oscillation frequency: 1.7 Hz (100 cycles per minute)
- amplitude of oscillation: 0.5° of arc (An amplitude of oscillation of 1° of arc is permitted as an alternative. If such an amplitude is chosen, measure t_{s2} instead of t_{s1})
- torque range: to be chosen to give at least 75 percent of full-scale deflection at F_{max}
- die temperature: 160 °C ± 0.3 °C
- pre-heat time: None.

The two types of curemeter cannot give identical results.

7 EVALUATION OF MOONEY VISCOSITY OF TEST MIXES

Determine the mooney viscosity in accordance with IS 3660 (Part 7).

Record the result as ML (1 + 4) at 100 °C.

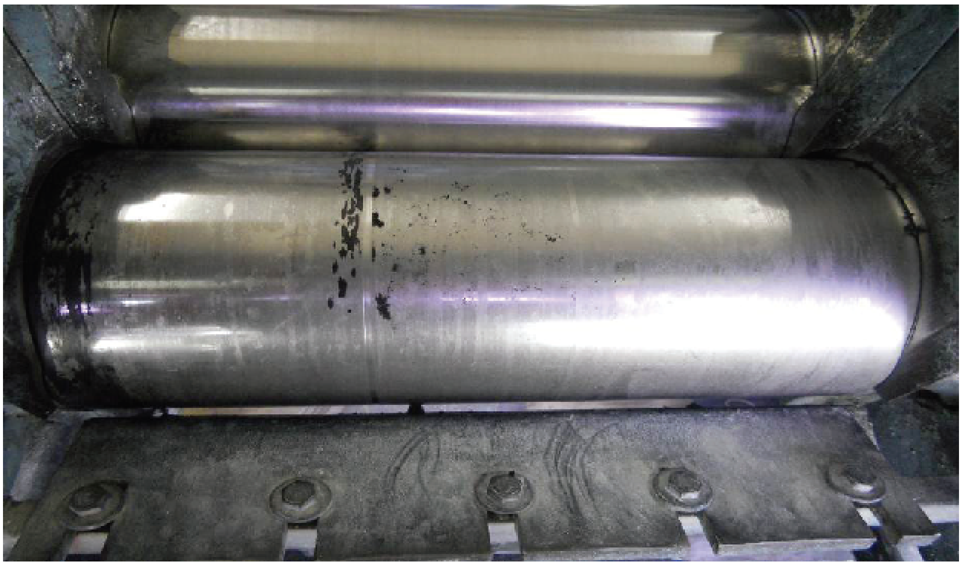

FIG.1 EXAMPLE OF SCRAPER BLADE

8 EVALUATION OF TENSILE STRESS-STRAIN PROPERTIES OF VULCANIZED TEST MIXES

WARNING — Formation of nitrosamines is possible during the cure.

Vulcanize sheets at 160 °C for 30 min.

Condition the vulcanized sheets/buttons for 16 h to 96 h at a standard laboratory temperature and, if possible, at a standard laboratory humidity as defined in ISO 23529.

Measure the stress-strain properties in accordance with IS 3400 (Part 1).

9 EVALUATION OF SHORE HARDNESS OF VULCANIZED TEST MIXES

Measure the Shore hardness using durometers with the A-scale for rubbers in the normal hardnessrange in accordance with IS 3400 (Part 23/Sec 1).

10 TEST REPORT

The test report shall include the following:

a) A reference to this Indian Standard (that is IS 16713);
b) All details necessary for the identification of the sample;
c) The method used for the ash determination [method A or B of IS 3708 (Part 9)];
d) The method used for the acetone extract determination (method A or method B of ISO 1407);
e) The method used for the carbon black determination (method A of ISO 1408);
f) The reference materials used to prepare the test mix;
g) The ambient conditions in the laboratory during preparation of the test mix;
h) The type of curemeter used, the defined time at which M_H was measured and the amplitude of oscillation used for the curemeter test, as defined in **6**;
j) Any unusual features noted during the determination;
k) Details of any operation not included in this Indian Standard or in the International Standardsto which reference is made, as well as of any operation regarded as optional;
m) The results and the units in which they have been expressed; and
n) The date of the test.

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Amendments Issued Since Publication

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